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CHLORINATION TASTES AND ODORS[1]

By Wellington Donaldson[2]

Following the court decree of May, 1910, in the well known Jersey City case, which was generally regarded as establishing the legitimacy of chlorination as a method of water treatment, in so far as concerned American practice, hypochlorite plants began to be installed for certain supplies experiencing difficulty in conforming to accepted standards for bacterial purity. By 1912 or 1913 the practice of sterilization by hypochlorite had become generally established. Later the marketing of successful apparatus for feeding liquid chlorine or chlorine gas gave great impetus to chlorination treatment, resulting in the displacement of many hypochlorite installations by liquid chlorine apparatus and extending marvelously the utility of this treatment both for permanent installations and for emergency and portable use. The outstanding example of the latter is the service with the various armies in the Great War. Whether or not one agrees with the dictum that no "water supply is safe without chlorination"—and the writer personally finds some objection to that phrase—it cannot be disputed that unchlorinated water supplies derived from surface sources are much in the minority in the United States at the present time. The marked decline in typhoid incidence and mortality in the principal urban communities testifies strongly to the wisdom of the practice.

Concurrent with the general adoption of chlorination there began to be noted an annoying prevalence of tastes and odors in many supplies so treated. During the ensuing decade, 1912-1922, a great deal has been done towards eliminating tastes and odors, although unfortunatly at the present time complaints are not infrequent even with some of the larger water supplies kept under close technical supervision. Many of the troubles of the earlier hypochlorite installations were unquestionably due to lack of adequate devices for feeding the solution at a uniform rate or in proportion to the water treated, to the failure to determine on a routine basis the actual strength of the chemical, or to the failure to make tests for residual chlorine on the treated water. There are today numerous hypochlorite installations 8, 10 or even 12 years old, in daily service with satisfactory results both as to sterilization and comparative freedom from tastes and odors, but the considerable supplanting of the older method by liquid chlorine has undoubtedly resulted in elimination of much taste complaint, by reason of the obvious advantages of liquid chlorine in the way of close regulation, uniform strength of chemical, ease of adjustment, etc. Even so, it is by no means rare to find liquid chlorine installations where proper attention is not given to making the doses follow closely the variations in water flow and the variations in quality of the water treated. It is not surprising, then, that such installations give trouble occasionally on account of overdosing.

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[1] Read before Chemical and Bacteriological Section, Philadelphia Convention, May 19, 1922.

[2] With Fuller and McClintock, New York, N. Y.

It is a necessary preliminary to any study of tastes and odors to ascertain if the taste and odor are absent from the unchlorinated water. If taste and odor are found only in the chlorinated water, their occurrence may be attributed to one or more of the following causes:

a. Excess or "free" chlorine.

b. Substitution compounds of chlorine with organic substances.

c. Killing of organisms with subsequent liberation of aromatic substances, which may or may not unite with chlorine.

With filtration plants delivering directly into the distribution system the last factor may be eliminated.

The taste of chlorine itself, if present in considerable excess, is easy to recognize by comparing with very weak solutions of chlorine or bleach. In the writer's experience 0.4 ppm., shown as "residual" chlorine, is about the limit of the average person's ability to taste pure chlorine, though some persons with sensitive taste can detect a lesser amount.

The absence of taste after boiling has been suggested as a means of differentiating free chlorine from its substitution compounds, but is not entirely reliable, as some substitution products are largely removed by a few minutes boiling. The addition of an "anti-chlor" such as sodium thiosulphate, to the sample before testing has been found useful in deciding as to the possibility of free chlorine being a factor. In connection with the possibility of excess chlorine the "chlorine absorption" value advocated by Race, Wolman and others (1, 2, 6, 7) will be found useful. Many water purification plants control chlorine feeding on the basis of maintaining a fixed"residual" chlorine of 0.1 to 0.3 ppm. in the effluent, the desired amount being determined by experience for that particular supply. The Wolman method, by reason of establishing a standard dose and a standard contact period, has the value of an exact laboratory expression, which makes all results comparable, but the writer prefers for practical plant operation the second method, which involves only a simple color text with o-tolidin and the matching against bottle standards (the writer uses 4-oz. oil sample bottles, E. & A. no. 1062, in preference to Nessler tubes)

It is usually the case that odors and tastes are much more pronounced at points on the distribution system than at the purification plant, and it is rare that tests for residual chlorine can be gotten from the consumer's faucet, by reason of rapid dissipation of the chlorine in contact with the surface of iron pipe.

If the chlorinated water gives no test for residual chlorine but has a disagreeable taste, it seems prima facie evidence that the trouble is due to "substitution" compounds, meaning thereby a combination of chlorine with some organic constituent of the water. Since the early days of chlorination there has been recognized the probability of organic matter combining with chlorine and thus giving rise to disagreeable tastes. The term "organic matter" as applied to water is a very broad term which includes many substances of whose nature we have little specific knowledge and at present little means of differentiating by laboratory tests.

The responsibility of humus substances from decaying vegetation for production of tastes and odors after chlorination is not very well defined. There are numerous examples of well-managed water supplies located on streams known not to receive any industrial wastes, yet troubled with chlorination tastes and odors. The correlation between the oxygen absorbed tests and the chlorine requirement has been fairly well established, but information is lacking as to the effect of high organic matter on production of taste after chlorination. In fact, those supplies with very low organic content seem to experience the greatest trouble.

In regard to the effect of algae on production of tastes and odors the symposium recently conducted by the Engineering News-Record (8) developed a lack of information bearing on this phase. The writer believes that algae frequently are responsible for mild chlorination taste and recalls an instance where Lyngbya, one of the blue-green algae, when allowed to collect in quantities on the filter beds, gave rise to an unpleasant taste in the water after chlorination. The taste was quite perceptible at the plant, though no complaints were reported by consumers. On the other hand, the writer has chlorinated an unfiltered supply heavily infested with diatoms (Cyclotella, principally) without appreciably altering the distinct "woody" taste present in the unchlorinated water.

The effect of protozoa on production of taste after chlorination, is fresh in the mind in connection with recent experiences of the New York Water Department reported by Brush (12) in dealing with synura in company with certain diatoms. The remarkable feature of the New York experience was the discovery that while the usual dose of chlorine accentuated the taste and odor from synura an increased dose eliminated the trouble. The same phenomenon had been previously reported by Girvan (4) and Houston (5) in connection with the New River supply of the London Metropolitan District.

The increasing pollution of our streams and waterways by industrial wastes has brought to light within the last three years another class of organic substances which affect very greatly the tastes and odors of chlorinated supplies. Principal offenders are the coke byproducts plants, city gas plants, producer gas plants and similar industries which discharge spent still liquors or even crude tars. The wastes from coal distillation contain phenol itself, but usually its homologs, such as the cresols, predominate. All of these substances exhibit a marked tendency to form "substitution" compounds with chlorine even in extremely high dilutions, resulting in an obnoxious medicinal taste in water supplies. The taste and odor are greatly intensified by heat.

Abundant instances are at hand bearing on the subject. The Milwaukee experiences with obnoxious tastes in its water supply were very clearly traced to the operation of certain coal distillation plants discharging wastes into the river and the lake (3). The writer has previously recorded (10) an experience with freshly dipped cast iron pipe. Hechmer (11) has presented evidence of the same trouble from use of freshly applied coal tar paint to an elevated water tank. Rowe (13) has naively and most interestingly reported an experience with pipe coating. The evidence is conclusive that the presence of phenols in water results, after chlorination, in greatly intensified and disagreeable tastes and odors. The numerous sources of phenol pollution to our streams makes the situation from the waterworks standpoint quite serious. So widespread is the trouble from this source that the writer is tempted to say that where a water supply experiences disagreeable "medicinal" tastes after chlorination, and tests show the absence of "free" or "residual" chlorine, then the trouble is apt to be due to the presence in the supply of phenols which may be detected and measured by suitable methods discussed below.

One of the simplest tests for phenol is that largely used in the Milwaukee investigation, namely, of simply adding to the suspected water a few drops of bleach solution and noting if the intense substitution taste occurs, particularly after warming. Such a test may not detect very minute traces of phenol, which however may affect the water supply. Usually some concentration method is necessary. In some early investigations at New Castle, Pa. the writer used successfully a method furnished by Prof. Aschmann of Pittsburgh, namely, skaking with ether in a separatory funnel, allowing the ether portion to evaporate and noting the residual odor. Adding to the residue a few cc. of distilled water and a drop of bromine water results in a turbidity or white precipitate of tri-bromphenol in the presence of appreciable quantities of phenol.

Shaking large volumes of water (1 to 20 liters) with chloroform and evaporating the settled chloroform in a covered dish was found simpler and more sensitive then the foregoing. Addition of a few drops of bleach solution just after the chloroform evaporated produced the characteristic medicinal tri-chloro-phenol odor.

In the New Castle investigations referred to interesting results were gotten by freezing, at one of the local ice plants, 400 pound cakes from the city water, which had at that time a disagreeable medicinal taste. It was found that the cores of the cakes had a pronounced and unmistakable odor and taste of cresol. The engineer of the ice-plant promptly called it a "lysol" odor, which is quite descriptive. On melting the core ice the cresol odor disappeared and was replaced by a strong woody odor.

The simple qualitative tests mentioned, though useful in helping decide the nature of the polluting substances, have little or no value for quantitative study. E. P. Fager, then chemist of the water company at South Pittsburgh, Pa., in studying in 1919 a variety of methods, with a view of quantitative estimation, devised a modification of the Folin-Denis reagent suitable for estimation of phenols in water. The method with slight modification was later used in the New Castle investigation in the winter of 1919–20. Following Scott's publication of the method (14), successful results from the method have been reported from a number of laboratories. The Folin-Denis reagent is quite sensitive to phenols (about 0.1 ppm. without distillation) although responsive also to quite a number of other substances, as various observers have noted. The different color reactions, varying from blue to green, are not without value in distinguishing phenol from some of its homologs. The reagent responds also to the higher phenols from the wood distillation industry.

It may be interesting to record here the findings at the New Castle plant that water containing 0.3 ppm. phenol gave rise to noticeable taste after chlorination, which agrees closely with the 0.2 ppm. reported by Ellms and Lawrence (9) as a result of dilution experiments.

In connection with the trouble by some laboratories in getting distilled water blanks for this reagent the writer calls attention to the fact that distilled water prepared from a water supply containing phenols will certainly contain phenol also. Repeated distillation will not eliminate phenol unless a batch still is used and first portions, containing the bulk of phenols, discarded. The writer has found it much simpler to procure distilled water from a spring or other supply not contaminated with phenols.

Another sensitive quantitative test for phenols in water has been proposed by Fox and Gauge (17), based on the diazo reaction familiar to the organic chemist. Apparently this was used in connection with investigations of the Joint Fisheries Committee in England, as no mention was made of its use in connection with public water supplies. To save the trouble of referring to the English journal in which the article was published the following brief abstract is given:

> Place 100 cc. of sample in a Nessler jar; add 5 cc. sodium hydroxide (8 per cent); add 10 per cent diazotized sulphanilic acid.[3] On stirring, an orange color is formed when tar acids are present.

Distilling 250 to 500 cc. of sample is advised if the sample contains turbidity. The author claims a sensitiveness of less than 1 ppm.

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[3] The diazotized sulphanilic acid is prepared 5 minutes before use by mixing 5 volumes of sulphanilic acid (1.91 grams in 250 cc. of distilled water) with one volume sulphuric acid (1 to 3), adding 5 volumes sodium nitrite (0.85 gram in 250 cc. distilled water) and cooling in a stream of cold water.

in comparing with standards prepared by mixing the three cresols in stated proportions. Other observers report a sensitiveness of 0.1 ppm. to pure phenol.

In connection with quantitative determinations of phenols in water, the writer suggests that pure phenol, C_6H_5OH, be used as the standard expression for the analysis, whether the actual substance under test be phenol, cresol, guaiacol or some other phenolic substance. Such expression is comparable with the expression of alkalinity, incrustants and hardness in terms of calcium carbonate.

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